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(54) Vapour phase processing

(57) A bubbler as illustrated in Fig. 2 (11) for solid reactant sources (18) such as trimethylindium for use in chemical vapour deposition includes a gas port (14) and a dip tube (16) in a chamber, the dip tube extending substantially from one end of the chamber to the other. Carrier gas such as hydrogen enters the chamber (12) via the port (14) and the output gas, which also includes vapour from the solid reactant material (18), exits the chamber via the dip tube (16). This is the reverse flow direction to that employed in conventional bubblers and leads to a more stable output. To prevent the entrainment of solid particles the dip tube may be of a wide bore dimension and/or a filter (20) may be incorporated therein. The bubbler is immersed in a constant temperature water bath and the bath temperature determines the vapour pressure of the solid reactant source. In use with hydrogen as the carrier gas and trimethylindium (TMIn) as the solid reactant source the hydrogen concentrates at the top of the chamber since it is less dense than the H_2 – TMIn mixture formed as a saturated mixture in equilibrium with solid TMIn and will begin to force the H_2 – TMIn downwards.

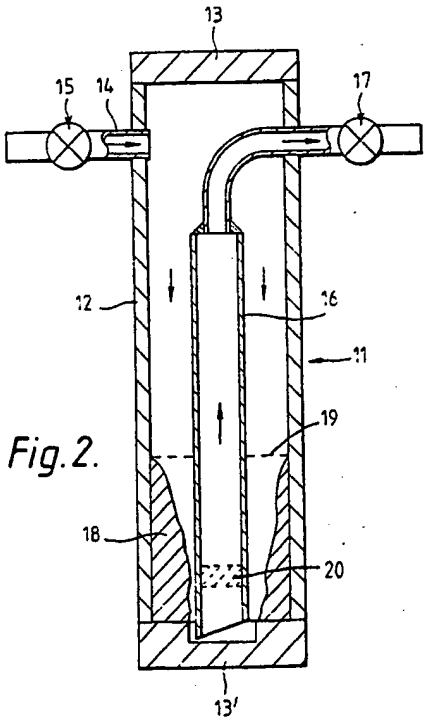

Fig. 2.

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At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

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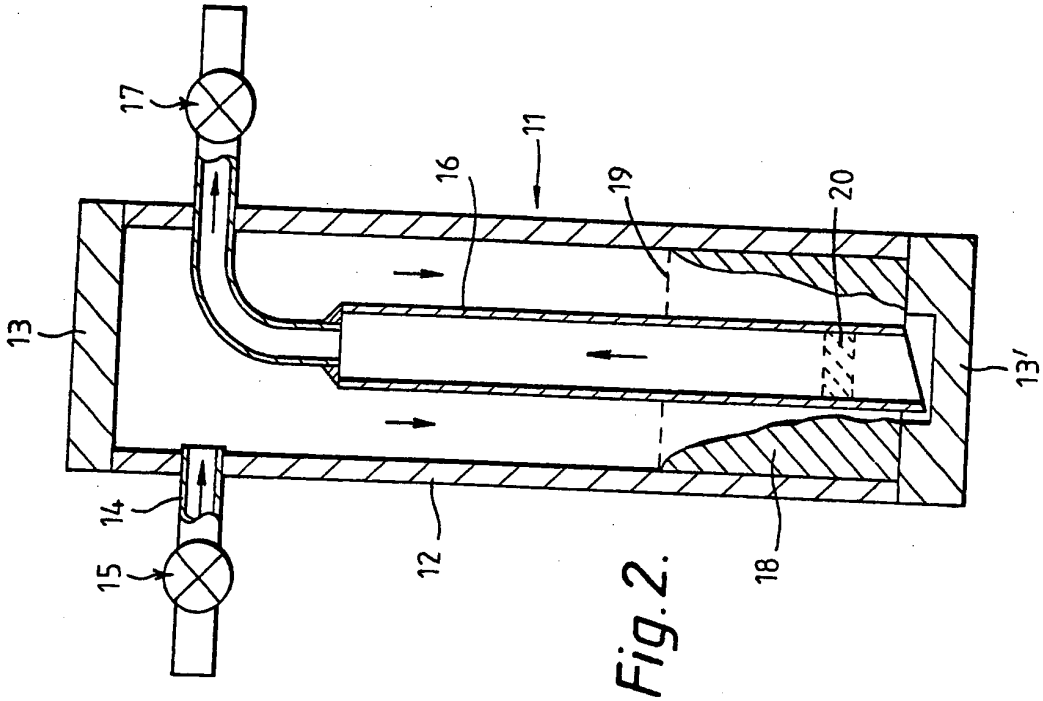

Fig.2.

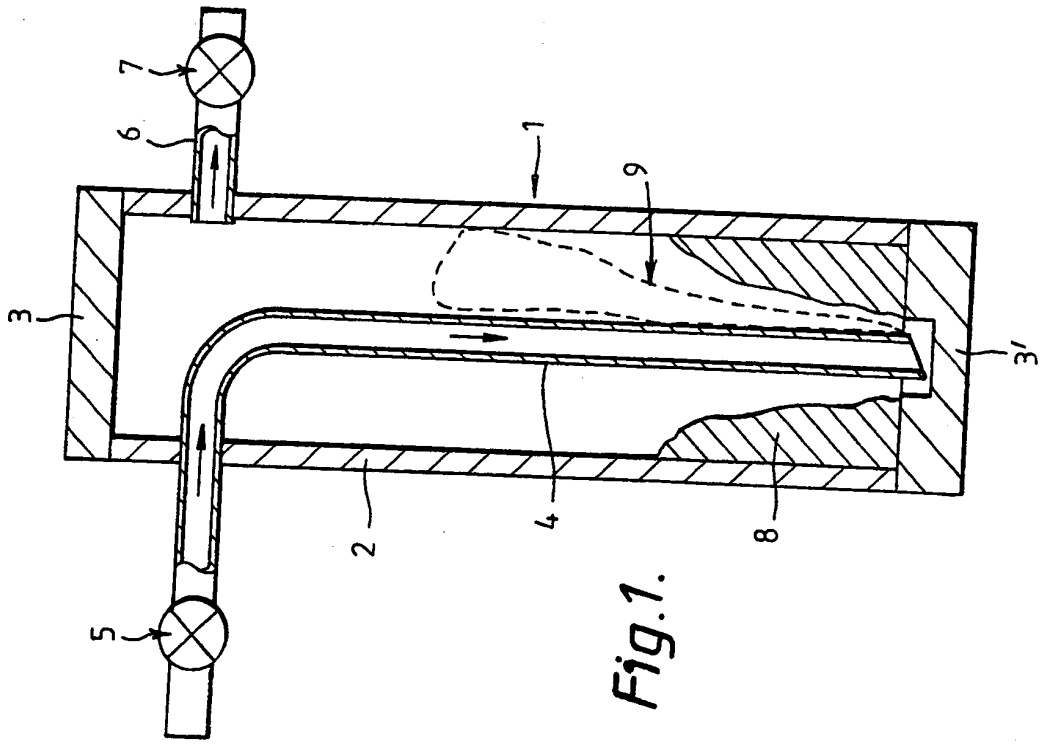

Fig.1.

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VAPOUR PHASE PROCESSING

This invention relates to vapour phase processing and in particular metal organic chemical vapour deposition (MOCVD) especially metal organic vapour phase epitaxy (MOVPE)

A conventional design of bubbler for solid metal organic reactant sources, such as trimethylindium (TMIn), is illustrated in Fig. 1. Trimethylindium is a solid whose melting point is about 88 Deg. C. The bubbler 1 is usually partially immersed in a constant temperature water bath, although alternatively it may be totally immersed. The bath temperature determines the TMIn vapour pressure and may be between 17 and about 60 Deg. C depending on the particular application. A temperature of 17 Deg. C would be used when the pipe work in the vapour phase epitaxy reactor was unheated, but for moderate flow rates a temperature of 40 to 50 Deg. C is a good compromise. The conventional bubbler 1 is made of stainless steel and comprises a cylinder 2 which is sealed at both ends 3, 3'. A dip tube 4 extends through the cylinder wall close to one end 3 of cylinder and towards the bottom of the cylinder at the other end 3'. A valve 5 is provided for shut-off purposes. An outlet tube 6 extends through the cylinder wall close to the one end 3, that is at the same end of the cylinder 2 as the dip tube 4. A valve 7 is provided for shut off purposes. In the case of a totally immersed bubbler the inlet and outlet arms may point 'upwards' rather than 'outwards' as drawn. Inside the cylinder 2 and extending from the end 3' is a deposit 8 of the solid reactant, for example TMIn. When the bubbler is loaded with TMIn, hydrogen is introduced into the cylinder and the valves

5 and 7 closed. As a result the bubbler will become filled with a uniform H_2 - TMIn mixture which is in equilibrium with the solid TMIn. The density of hydrogen saturated with TMIn at 40 Deg. C is approximately 0.130 g/litre, whereas the density of pure hydrogen at 40 Deg. C is approximately 0.079 g/litre, both at 760 torr. The corresponding figures at 50 Deg. C are 0.181 g/litre and 0.075 g/litre.

In use of the conventional bubbler, hydrogen is input via dip tube 4 and is intended to push the initial H_2 and TMIn mixture out of the cylinder via outlet tube 6, valves 5 and 7 being open, as well as to entrain further TMIn vapour. The input hydrogen emerging from the bottom of the dip tube 4 has a density considerably less than that of the surrounding hydrogen and TMIn mixture and it is considered that it will form a plume 9, which, very loosely, is analogous to smoke from the hot end of a cigarette, through the denser hydrogen - TMIn mixture. For the bubbler to behave in a stable fashion over many runs it is necessary for the hydrogen plume to be completely saturated with TMIn by the time it has risen to the top of the TMIn deposit 8. It is unlikely that this condition will be met in all cases, especially when much of the TMIn has been consumed. It is considered that the hydrogen and TMIn mixture at the top of the bubbler cylinder most probably will be hydrogen rich due to incomplete mixing in the hydrogen plume.

It is an object of the present invention to provide an alternative bubbler with improved performance to the aforementioned conventional bubbler.

According to one aspect of the present invention there is provided a bubbler, for solid reactant sources, including a chamber, a dip tube in the chamber extending substantially from one end of the chamber to the other end of the chamber, the solid

reactant being disposed in use of the bubbler at said other end of the chamber, and a gas port adjacent said one end of the chamber the gas port comprising an inlet part for a carrier gas, output gas exiting the chamber via the dip tube.

According to another aspect of the present invention there is provided a method of operating a bubbler for solid reactant sources, the bubbler including a chamber, a dip tube extending in the chamber substantially from one end of the chamber to the other end of the chamber and a gas port adjacent said one end of the chamber, solid reactant source material being disposed in the chamber at said other end thereof, the method including admitting a carrier gas into the chamber through said gas port and collecting output gas from the dip tube.

An embodiment of the invention will now be described with reference to Fig. 2 of the accompanying drawings which is partially in cross-section and partially schematic.

The bubbler 1) illustrated in Fig. 2 is made of stainless steel and comprises a cylinder 12 which is sealed at both ends 13, 13'. An inlet tube 14 extends through the cylinder wall close to end 13. A valve 15 is provided for shut-off purposes. An outlet dip tube 16 extends within cylinder 12 from adjacent to the end 13' and exits the cylinder through the cylinder wall 12 close to the end 13. The uppermost portion of the outlet tube 16 and that exiting the cylinder is of smaller diameter than the remainder thereof in the version illustrated. A valve 17 is provided for shut-off purposes. Inside the cylinder 12 and extending from the end 13' is a deposit 18 of solid reactant, for example, TMIn.

When the bubbler is loaded with the deposit 18, hydrogen is introduced into the cylinder 12 and the valves 15 and 17 are closed. As a result the bubbler will become filled with a uniform H_2 - TMIn mixture which is in equilibrium with the solid TMIn, and thus a saturated mixture.

When the bubbler is in use, hydrogen is introduced via inlet 14 and it concentrates at the top of the cylinder 12 adjacent to end 13, since it is less dense than the H_2 - TMIn mixture, and will begin to force the H_2 - TMIn mixture downwards. There will be a plane stable interface between the hydrogen and the H_2 - TMIn mixture. When this interface reaches the top of the TMIn deposit (indicated by dashed line 19) the hydrogen side will begin to saturate with TMIn. At the line 19 there is thus a transition between hydrogen and a H_2 - TMIn mixture. If the hydrogen input flow rate is too great there will be insufficient time for saturation to occur. In this case it will be necessary to raise the temperature of the TMIn, by raising the water bath temperature, and reduce the flow of hydrogen so that the same number of molecules of TMIn per minute leave the bubbler. If the hydrogen flow is high there is a possibility that particles of solid TMIn may be entrained in the gas stream and swept up the outlet (dip) tube 16 into the subsequent heated pipework (not shown) where they would increase the TMIn content of the output. To reduce this risk the diameter of the initial part of the tube 1) through which the gas flows is increased in comparison with the remainder so that the upward flow velocity of the gas is reduced and particles can fall back under gravity. Alternatively or additionally, an inlet filter 20 indicated in dashed lines may be incorporated in or at the end of the dip

tube, which may be of the smaller or wider dimensions. A fairly coarse filter is envisaged, say 5-10 μm. The inlet and outlet tubes are generally of the order of 0.25" OD. The wider section of the dip tube 16 may be of the order of 0.50" OD. The velocity of the gas in an 0.50" tube at 50 cm^3/min is less than 1 to 2 cm/sec. The tube 16 may alternatively have a widened central section and narrow end sections in order to achieve flow velocity reduction.

As will be appreciated the hydrogen flow through the bubbler of Fig.2 is reversed in comparison with Fig.1. This however enables a more stable situation to be achieved. The input light hydrogen fills the entire top of the cylinder and its density increases progressively by picking up TMIn from the solid deposit until it enters the dip tube 16. In the conventional bubbler there is the plume of hydrogen, whose shape depends on the geometry of the bubbler, the disposition of the TMIn and the flow rates, and a less stable and more uncontrollable situation pertains.

Whereas the bubbler has been described is of stainless steel other inert materials such as glass may be employed. Whereas the carrier gas has been described as hydrogen it could alternatively be helium or another carrier gas whose density is significantly less than the density of the mixture of the vapour from the solid to be transported and the carrier gas.

CLAIMS:-

1. A bubbler, for solid reactant sources, including a chamber, a dip tube in the chamber extending substantially from one end of the chamber to the other end of the chamber, the solid reactant source being disposed in use of the bubbler at said other end of the chamber, and a gas port adjacent said one end of the chamber, the gas port comprising an inlet port for a carrier gas, output gas exiting the chamber via the dip tube.

2. A bubbler as claimed in claim 1 wherein the dip tube includes a portion which is of a wider section than the remainder thereof whereby to reduce the flow velocity of the gas in said portion and prevent the entrainment of particles of the solid reactant.

3. A bubbler as claimed in claim 1 or claim 2 including an inert filter in the dip tube.

4. A bubbler as claimed in any of the preceding claims and manufactured of stainless steel.

5. A bubbler as claimed in any of the claims 1 to 3 and manufactured of glass.

6. A bubbler as claimed in any of the preceding claims and used with a carrier gas whose density is significantly less than the density of the mixture of the vapour from the solid reactant source and the carrier gas.

7. A bubbler as claimed in claim 6 wherein the solid reactant source is TMIn and the carrier gas is hydrogen.

8. A method of operating a bubbler for solid reactant sources, the bubbler including a chamber, a dip tube extending in the chamber substantially from one end

of the chamber to the other end of the chamber and a gas port adjacent said one end of the chamber, solid reactant source material being disposed in the chamber at said other end thereof, the method including admitting a carrier gas into the chamber through said gas port and collecting output gas from the dip tube.

9. A method as claimed in claim 8 wherein the density of the carrier gas is significantly less than the density of the mixture of the vapour from the solid reactant source and the carrier gas.

10. A method as claimed in claim 9 wherein the solid reactant source is TMIn and the carrier gas is hydrogen.

11. A bubbler substantially as herein described with reference to and as illustrated in Fig. 2 of the accompanying drawings.

12. A method of operating a bubbler substantially as herein described with reference to Fig. 2 of the accompanying drawings.

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